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U1S S2297

(56) Documents Cited
GB 2000812 A US 4415421 A

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UK CL (Edition M) C7F FBAA FBAD FBAE FBAH FBAL FBAP FBAR FBAT FBAX FPCX FPDX FPEX
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ONLINE DATA BASES : WPI, CLAIMS

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(54) Golden colored part comprising coatings of titanium nitride and gold

(57) An ornamental golden colored part comprises a substrate and a coating, said coating comprising an inner layer of TiN or TiN alloy formed on said substrate, an outer layer of Au or Au alloy covering the surface of said substrate and an intermediate layer disposed between said inner and outer layers; wherein the intermediate layer is formed so that its composition changes from a mainly TiN or TiN alloy phase gradually to a mainly Au or Au alloy phase. The said gradually changing alternate layer may consist of four different phases which include: an "a phase" which include pure TiN or TiN as main component and other elements selected from Zr, Al, C, O; a "b phase" which include TiN as main component and Au or Au and other elements selected from Zr, Al, C, O, Cu, Ni; a "c phase" which include Au as main component and TiN or TiN and other elements selected from Zr, Al, C, O, Cu, Ni; and a "d phase" which include pure Au or Au as main component and Cu and/or Ni. The composition of said phases in the gradually changing alternate layer changes gradually. According to the present invention, the color and wear resistance of the plated part are better than the part plated with a single intermediate alloy layer comprising TiN and Au. This colored part may be produced by placing three sources of Ti or Ti alloy and one source of Au or Au alloy is a circular orbit; rotating the part around the centre of the chamber; turning on the 3 Ti sources; then turning on the Au source; and finally turning ff the Ti sources.

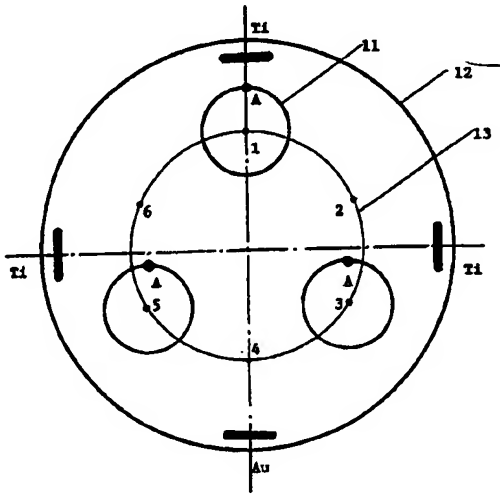

FIG 1

Claim 1

GB 2 284 431 A

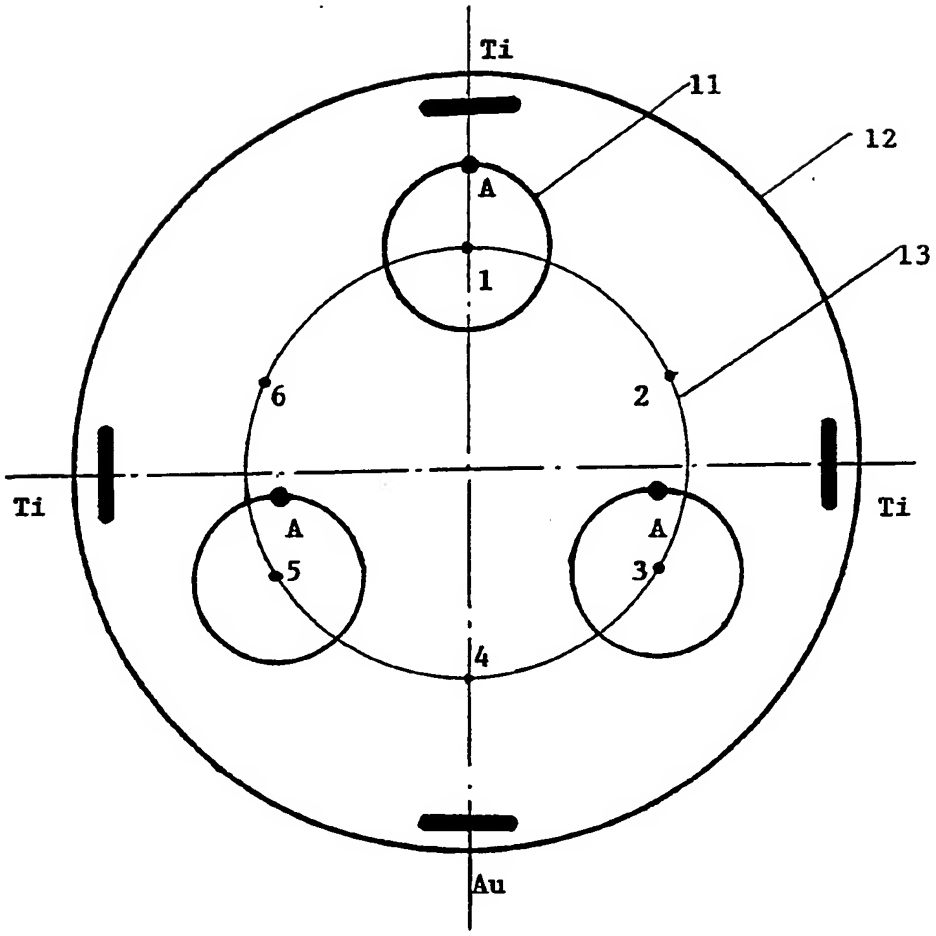

FIG 1

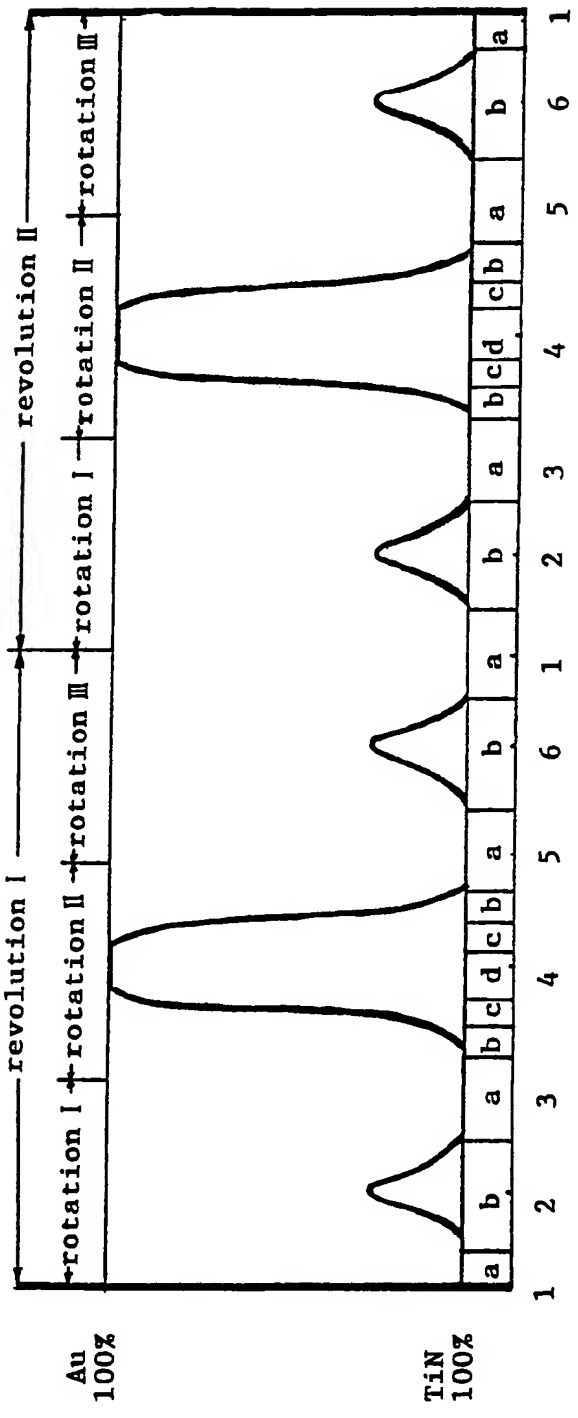
revolution I
revolution II
rotation I
rotation II
rotation III
rotation I
rotation II
rotation III
a b a b c d b c b a b a b c d b a b a
1 2 3 4 5 6 1 2 3 4 5 6 1
Au 100%
TiN 100%

FIG 2

GOLDEN COLORED PART

This invention relates to an ornamental golden colored part.

In order to produce golden colored ornamental parts, gold plating layer has been employed for many years. However, a gold plating layer exhibits poor wear resistance. For ensuring a long service life for such parts, the plated gold layer is required to have a thickness in excess of 10 microns. Since 1970, golden colored coating of titanium nitride (TiN) obtained by physical vapour deposition (PVD) has been utilized for making gold colored ornamental parts. TiN is a super-hard material and exhibits high corrosion resistance and wear resistance. When TiN is deposited as a coating layer the thickness of the plated gold layer can be reduced down to 0.5 micron. Unfortunately, the golden color of TiN coating is significantly different from that of natural gold. A layer of gold can be deposited on the surface of TiN coating layer and therefore to produce ornamental parts having a color tone of natural gold. However, when the gold layer is worn during the use of the ornamental parts, the TiN layer below appears and presents a different color in apparent contrast, so the ornamental quality of the parts is inferior.

To remedy this problem the addition of an intermediate layer between the TiN layer and the gold layer has been employed. The U.S. Patent No. 4,252,862

suggests a coating consisting of three layers in which an intermediate mixture phase layer is deposited between a TiN layer and an outermost gold layer. This additional intermediate mixture phase layer includes TiN and at least one element selected from the group consisting of 29 elements such as Au. The color tone and wear resistance of this coating is better than a TiN layer or a Au layer alone, respectively, but when the outermost gold coating layer becomes worn, the ornamental effect of the parts is still inferior.

According to one aspect of the invention, there is provided a part having a substrate and a golden colored coating thereon, said coating comprising an inner layer of TiN or TiN alloy formed on said substrate, an outer layer of Au or Au alloy covering the surface of said substrate and an intermediate layer disposed between said inner and outer layers, wherein the intermediate layer is formed in a way that its composition changes from a mainly TiN or TiN alloy phase gradually to a mainly Au or Au alloy phase.

Preferably the composition of the intermediate layer changes gradually on the surface of said substrate to form a plurality of sub-layers so that each said phase appears at least once in one said sub-layer across the thickness of said intermediate layer.

According to another aspect of the invention, there is provided a method of coating a part, comprising steps of arranging in a vacuum chamber at least a first

depositing source of mainly Ti or Ti alloy and a second depositing source of mainly Au or Au alloy, placing a part to be coated in said chamber and moving said part relatively to said first and second sources, introducing nitrogen gas into said chamber and actuating said first and second sources so as to form a coating layer of a composition gradually changing from a phase of mainly TiN or TiN alloy to a phase of mainly Au or Au alloy by said relative movement between said sources and the part.

Preferably said part and said first and second sources are arranged to have relatively revolving movement.

Preferably said first and second sources can be selectively actuated so as to form a further layer of mainly the same composition.

According to the present invention, when the outermost gold coating layer becomes worn, the gold phase which formed in the intermediate alternate layer will appear so as to produce a more uniform color tone and therefore desired ornamental effect of a part will maintain for a much longer period of time.

In another aspect of the present invention, there has been provided a golden colored part with a gradually changing alternate layer. The golden colored part comprises a substrate and a coating. The coating consists essentially of three layers, which include an innermost TiN layer or an alloy layer of TiN adjacent the substrate, an intermediate gradually changing

alternate layer on the side of above said innermost TiN layer and an outermost layer comprising Au or an Au alloy. The coating also can consist of four layers which include an innermost pure Ti layer or a Ti alloy layer adjacent to said substrate, an intermediate TiN layer or a TiN alloy layer on the side of said innermost Ti layer opposite from said substrate, a further intermediate gradually changing alternate layer positioned on the side of said intermediate TiN layer and an outermost layer comprising Au or an Au alloy based on Au with Cu and/or Ni.

The intermediate gradually changing alternate layer comprises four kinds of phases consisting of different compositions:

1. an "a phase" which is pure TiN alloy phase based on TiN with at least one element selected from a group consisting of Zr, Al, C and O;

2. a "b phase" i.e. TiN (Au) phase which is TiN alloy phase based on TiN with Au, or with Au and at least one element selected from the group consisting of Zr, Al, C, O, Cu and Ni;

3. a "c phase" i.e. Au (TiN) phase which is Au alloy phase based on Au with TiN or with TiN and at least one element selected from the group consisting of Zr, Al, C, O, Cu, and Ni;

4. a "d phase" which is pure Au or Au alloy based on Au with Cu and/or Ni.

Each of said four different kinds of phases

appears at least one time in the whole thickness of said gradually changing alternate layer with compositions of said phases changing gradually.

In another aspect of the present invention, there has been provided a method of preparing a golden colored part with gradually changing alternate layer. The method is that the cathode arc sources or magnetron sputtering sources are placed at equally intervals along the wall of a vacuum chamber therein for obtaining vapour phase therefrom. There are four groups of plating sources and the number of plating sources in every group is not limited. Among them, three groups of plating sources are pure Ti or a Ti alloy comprising Ti and Zr and/or Al. One group of plating sources is pure Au or an Au alloy comprising Au and Cu and/or Ni. The article to be plated is placed into the vacuum chamber and revolves round the centre of the vacuum chamber along a circular orbit as well as rotates on its own axis. The article rotates at least twice per revolution along the circular orbit.

For a more complete understanding of the present invention and for further objects and advantages thereof, reference may now be made to the following description taken in conjunction with the accompanying drawings in which:

Fig. 1 shows the deposition process of an embodiment according to the present invention.

Fig. 2 shows that the composition of the

gradually changing alternate layer of said embodiment according to the present invention.

Fig. 1 shows deposition process of an embodiment according to the present invention. The numeral 11 indicates an article to be plated, 12 indicates a vacuum chamber, 13 shows a circular orbit along which the article 11 revolves in the vacuum chamber 12. The numerals 1-6 as shown in Fig. 1 indicate different revolving and rotating locations of the point A on the article, and in Fig. 2 the compositions of the gradually changing alternate layer at different positions of the article are shown. The meaning of a,b,c and d, which represent different phases, have been described hereinabove. The Fig. 2 shows that the a,b,c and d four different phases are alternate and their compositions change gradually.

Example 1 (as shown in Fig. 1 and Fig. 2)

Four plating sources Ti and Au are placed into the vacuum chamber along the wall and are equally spaced, and three of them are mounted with pure Ti targets and the remaining one is with pure Au target. Thereafter, three plating sources of pure Ti are turned on when nitrogen gas is introduced into the vacuum chamber, so that a TiN plating layer is coated on the surface of the article. When the desired thickness, for example 0.5 microns of TiN layer is attained, the plating sources of both pure Ti and pure Au are turned on together so that a gradually changing alternate layer is coated on the TiN

layer. The ratio of revolution to rotation numbers of the article is 1 : 3.

If the point A on the article (as shown in Fig. 1) begins to revolve and rotate from the location as shown in Fig. 1 in this example, the structure of the gradually changing alternate layer is rather simple and only includes two components, i.e. TiN and Au. Fig. 2 shows a composition curve of the gradually changing alternate layer when the article revolves two circles along the orbit 13 and rotates six rotations at the same period of time. In Fig. 2, six Au peaks appear. After this gradually changing alternate layer is coated, the plating source of pure Ti is turned off. Therefore a thin layer of pure Au is coated on the surface of the article.

Example 2

There are four plating sources in the vacuum chamber, three of them are with a Ti alloy target comprising Ti and Al, and one of them is with an Au alloy target comprising Au and Cu. The nitrogen gas is introduced into the vacuum chamber. The ratio of revolution to rotation numbers of the article is 1 : 3. The components of the gradually changing alternate layer are TiN, Au and Al, Cu. The external color tone of the article formed is different from that of Example 1. Therefore, it may satisfy different consumer's needs for different color tones.

According to the present invention, the

uniformity of the color tone and the wear resistance of the coating with intermediate gradually changing alternate layer are significantly improved compared with a single alloy layer comprising TiN and Au etc. The TiN in gradually changing alternate layer is super-hard material and Au or an Au alloy is soft material. They are formed alternate so as to help each other. This is a kind of very good structure for wear resistance materials. Concerning the color tone, the pure gold layer included in the gradually changing alternate layer has a fine color tone of natural gold. In the gradually changing alternate layer, the composition between Au phase and TiN phase changes gradually. Accordingly it makes the adhesive force between Au and TiN phases stronger.

It is thus believed that the construction of the present invention will be apparent from the foregoing description. While the method and construction shown or described has been characterized as being preferred it will be obvious that various changes and modifications may be made therein.

CLAIMS:

1. A part having a substrate and a golden colored coating thereon, said coating comprising an inner layer of TiN or TiN alloy formed on said substrate, an outer layer of Au or Au alloy covering the surface of said substrate and an intermediate layer disposed between said inner and outer layers;

wherein the intermediate layer is formed so that its composition changes from a mainly TiN or TiN alloy phase gradually to a mainly Au or Au alloy phase.

2. A part of claim 1, wherein the composition of the intermediate layer changes gradually on the surface of said substrate to form a plurality of sub-layers so that each said phase appears at least once in one said sub-layer across the thickness of said intermediate layer.

3. A method of coating a part, comprising steps of:

(i) arranging in a vacuum chamber at least a first depositing source of mainly Ti or Ti alloy and a second depositing source of mainly Au or Au alloy;

(ii) placing a part in said chamber and moving said part relatively to said first and second sources;

(iii) introducing nitrogen gas into said chamber; and

(iv) actuating said first and second sources so as to form a depositing layer of a composition

gradually changing from a phase of mainly TiN or TiN alloy to a phase of mainly Au or Au alloy by said relative movement between said sources and the part.

4. A method of claim 3, wherein said part and said first and second sources are arranged to have relatively revolving movement.

5. A method of claim 3 or claim 4, wherein said first and second sources can be selectively actuated so as to form a further layer of mainly the same composition.

6. A golden colored part comprising a substrate and a coating consisting essentially of three layers, an innermost TiN layer or TiN alloy layer adjacent to said substrate, an intermediate gradually changing alternate layer on the side of said innermost TiN layer or said TiN alloy layer and an outermost layer comprising Au or an Au alloy layer;

said intermediate gradually changing alternate layer comprised of four kinds of phases consisting of different composition:

1. an "a phase" which is pure TiN phase or TiN alloy phase based on TiN with at least one element selected from the group consisting of Zr, Al, C, and O;

2. a "b phase" i.e. TiN (Au) phase which is TiN alloy phase based on TiN with Au, or with Au and at least

one element selected from the group consisting of Zr, Al, C, O, Cu, and Ni;

3. a "c phase" i.e. Au (TiN) phase which is Au alloy phase based on Au with TiN or with TiN and at least one element selected from the group consisting of Zr, al, C, O, Cu, and Ni;

4. a "d phase" which is pure Au or Au alloy based on Au with Cu and/or Ni;

each of said four different phases appearing at least one time in the whole thickness of said gradually changing alternate layer with the compositions of said phases changing gradually.

7. A golden colored part according to claim 1 wherein said coating consisting essentially of four layers, an innermost pure Ti layer or a Ti alloy layer adjacent to said substrate, an intermediate TiN layer or a TiN alloy layer on the side of said innermost Ti layer or said Ti alloy layer opposite from said substrate, an intermediate gradually changing alternate layer positioned on the said first intermediate TiN layer or said TiN alloy layer and an outermost layer comprising Au or an Au alloy based on Au with Cu and/or Ni.

8. A method of preparing a golden colored part including the steps of:

placing four groups of plating sources along the wall of a vacuum chamber equally spaced, wherein three of

the four sources are pure Ti or a Ti alloy comprising Ti and Zr and/or Au, and the remaining one is pure Au or an Au alloy comprising Au and Cu and/or Zr, for obtaining variable composition in the golden colored part;

placing a part to be plated into the vacuum chamber and making said part revolving round the centre of the vacuum chamber along a circular orbit and rotating on its axis at the same time;

turning on said three pure Ti or Ti alloy sources and introducing nitrogen gas into the vacuum chamber for forming an innermost layer comprising TiN or TiN alloy adjacent to a substrate of the part; and

then turning on the Au source and making said part rotating at least twice per revolution for forming an intermediate gradually changing alternate layer on the side of said innermost TiN layer or TiN alloy layer opposite from said substrate; and

turning off the pure Ti sources or Ti alloy sources for forming an outermost layer comprising Au or Au alloy layer.

9. A method of preparing a golden colored part including the steps of:

placing four groups of plating sources along the wall of a vacuum chamber equally spaced, wherein three of the four sources are pure Ti or a Ti alloy comprising Ti and Zr and/or Al, and the remaining one is pure Au or an Au alloy comprising Au and Cu and/or Zr, for obtaining

variable composition in the golden colored part;

placing a part to be plated into the vacuum chamber and making said article revolving round the centre of the vacuum chamber along a circular orbit and rotating on its axis at the same time;

turning on said three pure Ti or Ti alloy sources for forming an innermost pure Ti layer or a Ti alloy layer adjacent to a substrate of the part;

introducing nitrogen gas into the vacuum chamber for forming a first intermediate TiN layer or a TiN alloy layer on the side of said innermost Ti layer opposite from said substrate; and

then turning on the Al source and making said part rotating at least twice per revolution for forming a second intermediate layer that is gradually changing alternate layer on the side of the first intermediate Ti layer or a TiN alloy layer, and turning off the pure Ti sources or Ti alloy sources for forming an outermost Au layer or Au alloy deposited on the gradually changing alternate layer.

10. A part having a surface formed substantially as described herein with reference to the accompanying drawings.

11. A method of preparing a part performed substantially as described herein with reference to the accompanying drawings.

Amendments t the claims have b en fil d as f llows

1. A golden colored part comprising a substrate and a coating consisting essentially of three layers, an innermost TiN layer or TiN alloy layer adjacent to said substrate, an intermediate gradually changing alternate layer on the side of said innermost TiN layer or said TiN alloy layer and an outermost layer comprising Au or an Au alloy layer;

said intermediate gradually changing alternate layer comprised of four kinds of phases consisting of different composition:

1. an "a phase" which is pure TiN phase or TiN alloy phase based on TiN with at least one element selected from the group consisting of Zr, Al, C, and O;
2. a"b phase" i.e. TiN (Au) phase which is TiN alloy phase based on TiN with Au, or with Au and at least one element selected form the group consisting of Zr, Al, C, O, Cu, and Ni;
3. a "c phase" i.e. Au (TiN) phase which is Au alloy phase based on Au with TiN or with TiN and at least one element selected from the group consisting of Zr, Al, C, O, Cu, and Ni;

4. a "d phase" which is pure Au or Au alloy based on Au with Cu and/or Ni;

each of said four different phases appearing at least one time in the whole thickness of said gradually changing alternate layer with the compositions of said phases changing gradually, wherein the Au or Au alloy appears more than one time in the whole thickness of the coating.

2. A golden colored part according to claim 1 wherein said coating consisting essentially of four layers, an innermost pure Ti layer or a Ti alloy layer adjacent to said substrate, an intermediate TiN layer or a TiN alloy layer on the side of said innermost Ti layer or said Ti alloy layer opposite from said substrate, an intermediate gradually changing alternate layer positioned on the said first intermediate TiN layer or said TiN alloy layer and an outermost layer comprising Au or an Au alloy based on Au with Cu and/or Ni.

3. A method of preparing a golden colored part including the steps of:

Placing four groups of plating sources along the wall of a vacuum chamber equally spaced, wherein three of the four sources are pure Ti or a Ti alloy comprising Ti and Zr and/or Au, and the remaining one is pure Au or an Au alloy comprising Au and Cu and/or Zr, for obtaining variable composition in the golden colored part;

placing an article to be plated into the vacuum chamber and making said article revolving round the

centre of the vacuum chamber along a circular orbit and rotating on its axis at the same time;

turning on said three pure Ti or Ti alloy sources and introducing nitrogen gas into the vacuum chamber for forming an innermost layer comprising TiN or TiN alloy adjacent to a substrate of the article; and

then turning on the Au source and making said article rotating at least twice per revolution for forming an intermediate gradually changing alternate layer on the side of said innermost TiN layer or TiN alloy layer opposite from said substrate; and

turning off the pure Ti sources or Ti alloy sources for forming an outermost layer comprising Au or Au alloy layer.

4. A method of preparing a golden colored part including the steps of:

placing four groups of plating sources along the wall of a vacuum chamber equally spaced, wherein three of the four sources are pure Ti or a Ti alloy comprising Ti and Zr and/or Al, and the remaining one is pure Au or an Au alloy comprising Au and Cu and/or Zr, for obtaining variable composition in the golden colored part;

placing an article to be plated into the vacuum chamber and making said article revolving round the centre of the vacuum chamber along a circular orbit and rotating on its axis at the same time;

turning on said three pure Ti or Ti alloy sources for forming an innermost pure Ti layer or a Ti alloy layer adjacent to a substrate of the article;

introducing nitrogen gas into the vacuum chamber for forming a first intermediate TiN layer or a TiN alloy layer on the side of said innermost Ti layer opposite from said substrate; and

then turning on the Au source and making said article rotating at least twice per revolution for forming a second intermediate layer that is gradually changing alternate layer on the side of the first intermediate Ti layer or a TiN alloy layer, and turning off the pure Ti sources or Ti alloy sources for forming an outermost Au layer or Au alloy deposited on the gradually changing alternate layer.

5. A part having a surface formed substantially as described herein with reference to the accompanying drawings.

6. A method of preparing a part performed substantially as described herein with reference to the accompanying drawings.

Patents Act 1977 Examiner's report to the Comptroller under Section 17 (The Search report) 18	Application number GB 9400237.5
Relevant Technical Fields (i) UK Cl (Ed.M) C7F (FPCX,FPDX,FPEX,FBAA,FBAD,FBAE,FBAH,FBAL,FBAP,FBAR,FBAT,FBAX) (ii) Int Cl (Ed.5) C23C (14/00,14/06,14/14,14/16,14/18,14/22,28/00,28/02); G04B 37/22	Search Examiner P G Beddoe Date of completion of Search 17 February 1994
Databases (see below) (i) UK Patent Office collections of GB, EP, WO and US patent specifications. (ii) ONLINE DATABASES : WPI, CLAIMS	Documents considered relevant following a search in respect of Claims :- 1-11

Categories of documents

X:	Document indicating lack of novelty or of inventive step.	P:	Document published on or after the declared priority date but before the filing date of the present application.
Y:	Document indicating lack of inventive step if combined with one or more other documents of the same category.	E:	Patent document published on or after, but with priority date earlier than, the filing date of the present application.
A:	Document indicating technological background and/or state of the art.	&:	Member of the same patent family; corresponding document.

Category	Identity of document and relevant passages		Relevant to claim(s)
X	GB 2000812 A	(NOBUO NISHIDA) see especially page 3 lines 78-93	1,2
X	US 4415421	(CITIZEN WATCH) see especially Claim 1; Exs Table II	1-6

Databases:The UK Patent Office database comprises classified collections of GB, EP, WO and US patent specifications as outlined periodically in the Official Journal (Patents). The on-line databases considered for search are also listed periodically in the Official Journal (Patents).

DERWENT-ACC-NO: 1995-196185

DERWENT-WEEK: 199725

TITLE: Gold coloured part for ornaments, etc. - comprises titanium nitride lower layer, graded layer of four distinct phases and a gold@ outer layer

INVENTOR: WANG, D

PATENT-ASSIGNEE: CHANGCHENG TI-GOLD TECH COMBINED DEV COR[CHANN] , BEIJING GREAT-WALL TI-GOLD CORP[BEIJN]

PRIORITY-DATA: 1993CN-0120347 (December 4, 1993)

PATENT-FAMILY:

PUB-NO	PUB-DATE	LANGUAGE	PAGES	MAIN-IPC
GB 2284431 A	June 7, 1995	N/A	021	C23C 014/14
GB 2284431 B	June 4, 1997	N/A	000	C23C 014/14
CN 1089665 A	July 20, 1994	N/A	000	C23C 014/14

APPLICATION-DATA:

PUB-NO	APPL-DESCRIPTOR	APPL-NO	APPL-DATE
GB 2284431A	N/A	1994GB-0000237	January 7, 1994
GB 2284431B	N/A	1994GB-0000237	January 7, 1994
CN 1089665A	N/A	1993CN-0120347	December 4, 1993

INT-CL (IPC): B32B015/01, C23C014/14 , C23C014/34

ABSTRACTED-PUB-NO: GB 2284431A

BASIC-ABSTRACT:

The graded intermediate layer comprises: a first phase of TiN or TiN alloyed with Zr, Al, C and/or O; a second phase of TiN alloyed with Au and opt. Zr, Al, C, O, Cu and/or Ni; a third phase of Au alloyed with TiN and opt. Zr, Al, C, O, Cu and/or Ni; and a fourth phase of Au opt. alloyed with Cu and/or Ni. Each phase appears at least once in the graded layer with the boundaries between the phases graded, the Au (alloy) appearing at least twice in the entire coating. The part is formed by successive vacuum plating operations.

ADVANTAGE - Part has better wear resistance than a part with a single TiN/Au alloy intermediate layer.

ABSTRACTED-PUB-NO: GB 2284431B

EQUIVALENT-ABSTRACTS:

A golden coloured part comprising a substrate and a coating consisting essentially of three layers, namely an inner TiN or TiN alloy layer adjacent to the substrate, an intermediate compositionally modulated graded layer and an outer Au or Au alloy layer; the intermediate compositionally modulated graded layer comprising four kinds of sub-lay r composition, namely: a first sub-layer composition which is TiN phase, or TiN alloy with at least one of Zr, Al, C, and O; a s cond sub-layer composition which is TiN alloy phase based on TiN

with Au, or with Au and at least one of Zr, Al, C, O, Cu, and Ni; a third sub-layer composition which is Au alloy phase based on Au with TiN, or with TiN and at l ast one of Zr, Al, C, O, Cu, and Ni; a fourth sub-layer composition which is Au or Au alloy based on Au with Cu and/or Ni; ach of th four sub-layer compositions appearing at least one time in the intermediate compositionally modulated grad d layer, at least one said first, second or third sub-layer compositions being located between a said fourth sub-layer composition and the outer Au and Au alloy layer.

CHOSEN-DRAWING: Dwg.0/2 Dwg.1

TITLE-TERMS: GOLD COLOUR PART ORNAMENT COMPRISE TITANIUM NITRIDE LOWER LAYER GRADE LAYER FOUR DISTINCT PHASE GOLD@ OUTER LAYER

DERWENT-CLASS: M13 M26 P73

CPI-CODES: M13-F02;

SECONDARY-ACC-NO:
CPI Secondary Accession Numbers: C1995-090701